

AD-A049 558

HUGHES RESEARCH LABS MALIBU CALIF  
DIFFUSION PROCESS FOR FORMATION OF SINGLE-MODE WAVEGUIDE.(U)  
JAN 78 B CHEN

F/G 20/6

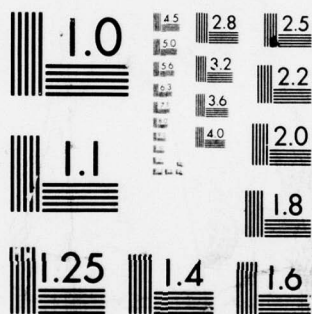
N00173-77-C-0138

NL

UNCLASSIFIED

| OF |  
AD  
A049558





MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS-1963-A

AD A 049558

AD NO. **JDC** FILE COPY

19

12  
B.S.

## DIFFUSION PROCESS FOR FORMATION OF SINGLE-MODE WAVEGUIDE

Bor-Uei Chen

Hughes Research Laboratories  
3011 Malibu Canyon Road  
Malibu, CA 90265

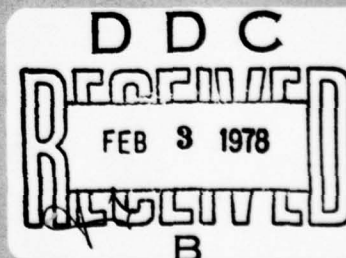
January 1978

Contract N00173-77-C-0138

For period 18 May 1977 through 18 December 1977

Approved for public release. Distribution unlimited.

Prepared for  
NAVAL RESEARCH LABORATORY  
4555 Overlook Avenue, S.W.  
Washington, DC 20375



UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) 6 DIFFUSION PROCESS FOR FORMATION OF SINGLE-MODE WAVEGUIDE		5. TYPE OF REPORT & PERIOD COVERED Final Report. 18 May 1977 - 18 Dec 1977
7. AUTHOR(s) 10 Bor-Wei/Chen		8. CONTRACT OR GRANT NUMBER(s) 15 N00173-77-C-0138
9. PERFORMING ORGANIZATION NAME AND ADDRESS Hughes Research Laboratories 3011 Malibu Canyon Road Malibu, CA 90265		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 12 34 p.
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Research Laboratory 4555 Overlook Ave., S.W. Washington, D.C. 20375		12. REPORT DATE 14 Jan 1978
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 29
		15. SECURITY CLASS. (of this report) Unclassified
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		15a. DECLASSIFICATION DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Integrated optics, Optical channel waveguide, CO <sub>2</sub> laser enhanced diffusion, Three dimensional channel horns		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The objective of this program is two-fold: (a) study of channel waveguide formation in LiNbO <sub>3</sub> and LiTaO <sub>3</sub> by Ti metal diffusion including Li <sub>2</sub> O out-diffusion suppression and lateral diffusion suppression; (b) development of three-dimensional channel waveguide horns designed to provide higher end-fire coupling efficiency to large-core single-mode fibers.		

DDC  
RECEIVED  
FEB 3 1978  
B

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

172 600

mt



UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

As a result of 1976 IR&D effort, we have developed a technique to eliminate  $\text{Li}_2\text{O}$  out-diffusion waveguide by annealing the crystal in  $\text{LiNbO}_3$  powder. Under this program, a series of experiments were carried out to study the temperature and time dependence of this powder treatment. Three significant results were obtained. First, it is confirmed that up to 1% molecular weight of  $\text{Li}_2\text{O}$  can be brought into the substrate surface layer through the powder treatment. Second, the compensation process ( $\text{Li}_2\text{O}$  in-diffusion) may occur at temperatures  $750^\circ\text{C}$  or lower. Third, 80% of the compensation process can be obtained in one-half hour when the annealing temperature is  $900^\circ\text{C}$ . The fast reaction rate implies that the powder treatment may not be dominated by the diffusion process. Solid-solid surface reaction may play an important role in this process.

Single-mode channel waveguides in  $\text{LiNbO}_3$  for this program were fabricated by diffusing Ti channels into Y-cut  $\text{LiNbO}_3$  wafers. The diffusion usually was carried out in the  $850$  to  $1000^\circ\text{C}$  temperature range in a flowing oxygen atmosphere for a period of 5 to 16 hours. The diffusion process proceeds in both lateral (Z-axis) and downward (Y-axis) directions. Because  $\text{LiNbO}_3$  has a uniaxial crystalline structure, it is of great importance to compare the diffusion rates along the Y and Z axes. In the course of this program, electron microprober was employed to measure the Ti concentration profile. The confinement of optical field in the diffused waveguide was also studied by profiling the near field mode pattern. Both measurements showed no sign of enhanced lateral diffusion.

A critical subject of utilizing integrated optic devices in optical communications and optical signal processing systems is the coupling between optical waveguide and single mode optical fiber. In view of the large index difference between modes in glass fiber ( $n \sim 1.50$ ) and in  $\text{LiNbO}_3$  channel waveguides ( $n \sim 2.21$ ), end-butt coupling seems a practical approach. High coupling efficiency is expected when the intensity distributions of the two waveguide modes are properly matched. The typical cross section of a single mode channel waveguide is about 4 by 2  $\mu\text{m}$ , which is much smaller than the single-mode fiber core diameter ranging from 5 to 15  $\mu\text{m}$ . To assure a good mode matching of the optical fields, one can use a three dimensional horn structure at the end of the channel guide. We have developed a technique for forming the three-dimensional horn structure; it uses a controlled  $\text{CO}_2$  laser heating technique. During the diffusion, a CW  $\text{CO}_2$  laser was focused at the end of the channel waveguide to raise the local surface temperature. To prevent the sample from cracking due to a large thermal gradient, the entire sample was maintained at temperatures ranging from  $700$  to  $850^\circ\text{C}$ . Because of the surface temperature difference, the diffusion proceeds faster in the laser heated area. Experimental results indicated an increase of 50% diffusion depth was obtained. However, the surface damage was always a problem for laser irradiation partially due to the fact that the diffusion was performed in air. The incorporation of an oxygen supply to the diffusion furnace is currently under way.  $\text{CO}_2$  laser enhanced diffusion will be carried out in a flowing oxygen atmosphere in the second phase of this program.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

# TABLE OF CONTENTS

SECTION		PAGE
I	Introduction and Summary	1
II	Diffusion Studies of $\text{Ti}:\text{LiNbO}_3$ Waveguide	4
	A. Channel Waveguide Formation	4
	B. $\text{Li}_2\text{O}$ Out-Diffusion Suppression	6
	1. $\text{SiO}$ and $\text{SiO}_2$ Encapsulated Ti Diffusion	6
	2. Temperature and Time Dependence of $\text{LiNbO}_3$ Powder Treatment	8
	3. A New Diffusion Source, Titanium Silica Film	12
	C. Lateral Diffusion Studies	14
III	Formation of Three-Dimensional Channel Waveguide Horns	19
IV	Suggested Plan for Phase II	24

ACCESSION for		
NTIS	White Section	<input checked="" type="checkbox"/>
DDC	Buff Section	<input type="checkbox"/>
UNANNOUNCED		<input type="checkbox"/>
JUSTIFICATION _____		
BY _____		
DISTRIBUTION/AVAILABILITY CODES		
Dist.	AVAIL. and/or	SPECIAL
A		

## FIGURES

	<u>PAGE</u>
1. Output mode of 4 $\mu\text{m}$ channel guide.	5
2. $\text{Li}_2\text{O}$ out-diffusion waveguide modes for a $\text{SiO}$ encapsulated Ti diffusion in $\text{LiNbO}_3$ .	7
3. $\text{Li}_2\text{O}$ out-diffusion waveguide modes for a $\text{SiO}_2$ encapsulated Ti diffusion in $\text{LiNbO}_3$ .	9
4. Modal index change for $\text{TE}_0$ mode as a function of temperature.	11
5. Modal index change as a function of annealing time for a fixed annealing temperature of $900^\circ\text{C}$ .	13
6. Single mode waveguide formed in $\text{LiNbO}_3$ by diffusing Ti doped silica.	15
7. Loss measurement for single mode $\text{LiNbO}_3$ waveguide fabricated by diffusing Ti doped silica.	16
8. Lateral diffusion profile of a Ti channel of 4 $\mu\text{m}$ initial width.	18
9. Near field mode pattern along the depth and width directions.	20, 21
10. Surface damage (a) after $\text{CO}_2$ laser enhanced diffusion and (b) after oxygen annealing.	25
11. Near field intensity distribution for (a) expanded and (b) unexpanded channel waveguides.	26
12. Intensity scan along the depth direction for (a) expanded and (b) unexpanded channel waveguides.	27
13. Intensity scan along the channel width direction for (a) expanded and (b) unexpanded channel waveguides.	28



## I. INTRODUCTION AND SUMMARY

The objective of this program is two-fold: (a) study of channel waveguide formation in  $\text{LiNbO}_3$  and  $\text{LiTaO}_3$  by Ti metal diffusion including  $\text{Li}_2\text{O}$  out-diffusion suppression and lateral diffusion suppression; (b) development of three dimensional channel waveguide horns designed to provide higher end-fire coupling efficiency to large core single mode fibers.

The fabrication of active channel waveguide devices (modulators, switches) compatible with single mode optical fibers is a key aspect of integrated optics technology. Low-loss optical waveguides fabricated in  $\text{LiNbO}_3$  and  $\text{LiTaO}_3$  crystals by in-diffusion of transition metal ions (Ti, Ni and V)<sup>1</sup> are very attractive for these applications. The excellent electro-optic and acousto-optic properties provide the capability for electrical control and high speed operation. Single mode channel waveguide circuits can be readily defined using well-developed photolithography techniques. However, a major problem in waveguide formation by metal in-diffusion is that at high diffusion temperature (850-1200°C), the crystalline substrate suffers the loss of loosely bound  $\text{Li}_2\text{O}$  through a surface out-diffusion process.<sup>2</sup> The deviation from crystal stoichiometry at the wafer surface increases the extraordinary refractive index. As a result, there is a planar waveguide formed due to  $\text{Li}_2\text{O}$  out-diffusion in addition to the waveguide formed by metal in-diffusion. The out-diffusion waveguide can confine a TE polarization wave propagating along the X-axis on a Y-cut wafer (or Y-axis on X-cut wafer). In a channel waveguide device, the existence of a planar out-diffusion waveguide would introduce excess cross-talk between channels. In addition, in an end-butt coupling configuration between a single mode optical fiber and a channel waveguide, a large portion of the optical energy goes to these unwanted out-diffusion modes which are excited by the optical fiber input. In an early experiment, we found that subsequent to the in-diffusion of Ti metal into Y-cut  $\text{LiNbO}_3$  the waveguide propagation caused by  $\text{Li}_2\text{O}$  out-diffusion can be eliminated by annealing the sample in  $\text{LiNbO}_3$  powder



at 900°C for 1 hour or longer.<sup>3</sup> The mechanism of this  $\text{Li}_2\text{O}$  compensation process is believed to be the following. At high temperature, the loosely bound lithium oxide molecules tend to escape from the surface. Since  $\text{LiNbO}_3$  powder has more surface area than  $\text{LiNbO}_3$  wafer, the  $\text{Li}_2\text{O}$  vapor pressure due to the powder is sufficient to build up a  $\text{Li}_2\text{O}$ -rich environment so that the out-diffusion process from the  $\text{LiNbO}_3$  wafer is suppressed. Furthermore, the compensation process of diffusion  $\text{Li}_2\text{O}$  back into a lithium deficient  $\text{LiNbO}_3$  wafer becomes thermodynamically favorable. It is a goal of this program to study in detail the temperature and time dependence of the  $\text{LiNbO}_3$  powder treatment. Under this program, a series of experiments was carried out on Y-cut  $\text{LiNbO}_3$  planar waveguides and three significant results were obtained. First, it is confirmed that up to 1% molecular weight of  $\text{Li}_2\text{O}$  can be brought into the substrate surface layer through the powder treatment. Second, the compensation process of  $\text{Li}_2\text{O}$  in-diffusion may occur at temperatures 750°C or lower. Third, 80% of the compensation process can be obtained in one-half hour when the annealing temperature is 900°C. The fast reaction rate implies that the powder treatment is not dominated by the diffusion process. Solid-solid surface reaction may play an important role in this process. This conclusion is evidenced by the fact that in-diffusion rate of  $\text{Li}_2\text{O}$  was slowed down when the sample was wrapped with platinum foil. When the  $\text{LiNbO}_3$  powder treatment was applied to the diffusion of Ti channels, the formation of single mode channel guides without  $\text{Li}_2\text{O}$  out-diffusion became difficult. Only two out of ten runs were successful. The process was so critical in temperature and time that either the suppression of  $\text{Li}_2\text{O}$  out-diffusion was incomplete or no channel guide was formed. It is believed that a thicker Ti coating ( $>200 \text{ \AA}$ ) and a lower powder annealing temperature are required to obtain truly single mode (no out diffusion modes) channel waveguides.

Single-mode channel waveguides in  $\text{LiNbO}_3$  for this program were fabricated by diffusing Ti channels into Y-cut  $\text{LiNbO}_3$  wafers. Lift-off technique was used to form the Ti diffusion pattern. The diffusion usually was carried out in the 850 to 1000°C temperature range in a flowing oxygen atmosphere for a period of 5 to 16 hours. The fundamental physical property of the diffusion process is that atoms tend to diffuse

from a region of high concentration to that of lower ones, at a rate proportional to the concentration gradient between the two regions. During the fabrication of the diffused channel waveguide, the diffusion process proceeds in both lateral (Z-axis) and downward (Y-axis) directions. Because  $\text{LiNbO}_3$  ( $\text{LiTaO}_3$ ) has a uniaxial crystalline structure, it is of great importance to compare the diffusion rates along the Y and Z axes. Particular interest was generated when an enhanced lateral diffusion, at a rate of about 20 times the downward diffusion rate, was reported in the experiment of diffusing Nb into  $\text{LiTaO}_3$ .<sup>4</sup> In this program, electron microprobe was employed to measure the Ti concentration profile. The confinement of optical field in the diffused waveguide was studied by profiling the near field mode pattern. Both measurements showed no sign of enhanced lateral diffusion.

A critical subject of utilizing integrated optic devices in optical communications and optical signal processing systems is the coupling between optical waveguide and single mode optical fiber. To date, most optically active devices are fabricated in Ti diffused  $\text{LiNbO}_3$  waveguides. In view of the large index difference between modes in glass fiber ( $n \sim 1.50$ ) and in  $\text{LiNbO}_3$  channel waveguides ( $n \sim 2.21$ ), end-butt coupling seems a practical approach.<sup>5</sup> The coupling is obtained with a cleaved optical fiber butt coupled (epoxied) against the polished edge of the channel waveguide. High coupling efficiency is expected when the intensity distributions of the two waveguide modes are properly matched. Usually, single-mode channel waveguides used in optical circuits require shallow waveguide depth to obtain a large interaction between guided optical field and the surface electrical field. The typical cross section of a single-mode channel waveguide is about 4 by 2  $\mu\text{m}$ , which is much smaller than the single-mode fiber core diameter ranging from 5 to 15  $\mu\text{m}$ . To assure a good mode matching of the optical fields, we can use either a small core fiber or a three dimensional horn structure at the end of the channel guide. Adopting a small core fiber is not practical because of the severe coupling tolerance requirements. On the contrary, the combination of three-dimensional horn structure and a relatively large core fiber will make the tolerance less critical. We have developed a technique for forming the three-dimensional horn structure; it uses a

controlled CO<sub>2</sub> laser heating technique. During the diffusion, a CW CO<sub>2</sub> laser was focused at the end of the channel waveguide to raise the local surface temperature. To prevent the sample from cracking due to a large thermal gradient, the entire sample was maintained at temperatures ranging from 700 to 850°C. Because of the surface temperature difference, the diffusion proceeds faster in the laser heated area. Experimental results indicated an increase of 50% diffusion depth was obtained. However, the surface damage was always a problem for laser irradiation partially due to the fact that the diffusion was performed in air. The incorporation of an oxygen supply to the diffusion furnace is currently underway. CO<sub>2</sub> laser enhanced diffusion will be carried out in a flowing oxygen atmosphere in the second phase of this program.

## II. DIFFUSION STUDIES OF Ti:LiNbO<sub>3</sub> WAVEGUIDES

### A. Channel Waveguide Formation

Single-mode channel waveguides were fabricated in Y-cut LiNbO<sub>3</sub> wafers by in-diffusion of titanium metal ions. The lift-off technique was used to form a Ti diffusion pattern. The pattern of 4 μm wide strips was first cut on the photoresist with conventional photolithography. After exposing and developing the photoresist, 200 Å Ti metal was e-beam deposited onto the photoresist pattern. The open area of the resist was thus filled with Ti metal; unwanted Ti on the photoresist was removed by dissolving the resist in acetone. The diffusions usually were carried out in the 850 to 1000°C temperature range in a flowing oxygen atmosphere for a period of 5 to 16 hours. Before diffusion, Ti metal had been oxidized at a relatively lower temperature, (for instance, 600°C). Waveguide loss of 4 μm wide channel guide was approximately on the order of 1 dB/cm. Figure 1 shows the single-mode structure coming out of a 4 μm channel guide through a rutile coupling prism.

The presence of Ti in the LiNbO<sub>3</sub> induces excess strain which causes difficulty in polishing the ends of the channel waveguides. There is always a strong tendency for the surface to chip around the channel ends. To avoid this problem, we worked on substrates with prepolished



7019-1

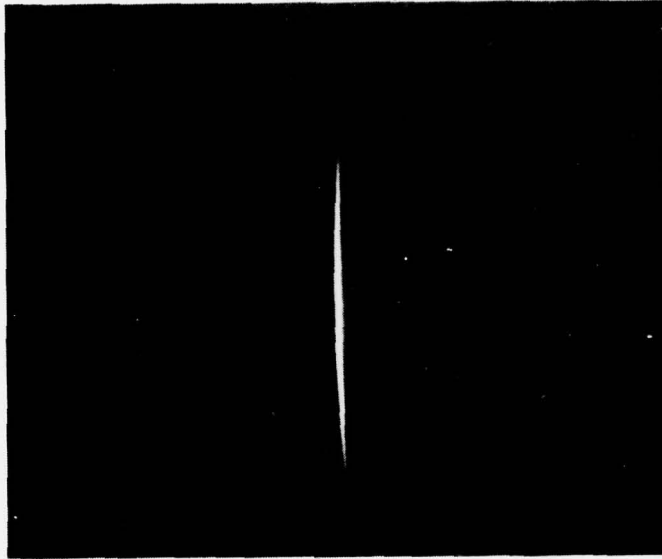


Figure 1. Output Mode of 4  $\mu\text{m}$  Channel Guide.



ends. In early experiments, the samples were processed using the spin-coating technique for applying the photoresist. As a result, the channel waveguides terminated before one edge of the substrate but were continuous to the opposite edge. By going to dip-coating of photoresist, channel waveguides were formed continuous to both ends of the substrate.

## B. $\text{Li}_2\text{O}$ Out-Diffusion Suppression

### 1. $\text{SiO}$ and $\text{SiO}_2$ Encapsulated Ti Diffusion

$\text{SiO}_2$  film has been widely used in semiconductor processing as diffusion masks because of its strong covalent bond structure. Recently, it was reported by Goldberg and Lee<sup>6</sup> that 500 Å thick  $\text{SiO}_2$  coating on  $\text{LiNbO}_3$  substrates can prevent  $\text{Li}_2\text{O}$  out-diffusion at high temperature. In their experiment, single mode channel waveguides (for TE polarization only) were formed by  $\text{Li}_2\text{O}$  out-diffusion through the open channels not masked by the  $\text{SiO}_2$  film. We have attempted to use  $\text{SiO}$  and  $\text{SiO}_2$  encapsulating films to prevent out-diffusion during the Ti diffusion process. However, both overcoating materials gave us negative results.

In the  $\text{SiO}$  encapsulated diffusion experiment, 200 Å Ti metal was first evaporated onto a Y-cut  $\text{LiNbO}_3$  substrate. Subsequently, a 1000 Å thick  $\text{SiO}$  overlayer was put down using an e-beam evaporation system. The  $\text{SiO}$  film was slightly brown in color. The diffusion was then carried out as usual. The diffusion temperature (T) and diffusion time (t) were  $T = 950^\circ\text{C}$  and  $t = 3\text{-}1/2$  h. The surface appearance of  $\text{SiO}$  coating showed no sign of deterioration after the high temperature diffusion process. However, the film became resistant to HF etching. There are four out-diffusion TE modes in addition to the single TE mode due to the Ti in-diffusion. (He-Ne laser was used to characterize the waveguide properties for all of the experiments reported here.) Figure 2 shows the waveguide mode pattern projected on a screen. The m-line on the left is the in-diffusion mode, while the four m-lines on the right are the results of  $\text{Li}_2\text{O}$  out-diffusion. The strong m-line scattering and intermode scattering shown in the figure indicates that the waveguide is very lossy. The modal index differential  $\Delta n$ , ( $\Delta n = n_{\text{eff}} - n_{\text{sub}}$ ), for different modes is listed below.

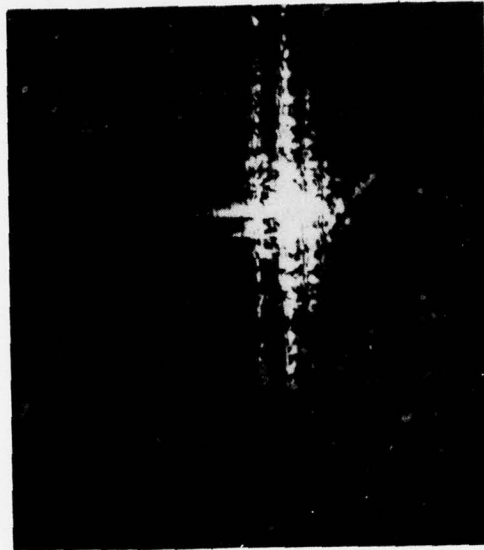


Figure 2.  $\text{Li}_2\text{O}$  Out-diffusion Waveguide Modes  
for a  $\text{SiO}_2$  Encapsulated Ti Diffusion  
in  $\text{LiNbO}_3$ .

mode	$\Delta n$
$TE_0$ (in-diffusion)	0.004
$TE_0$ (out-diffusion)	0.003
$TE_1$ (out-diffusion)	0.0018
$TE_2$ (out-diffusion)	0.0006
$TE_3$ (out-diffusion)	$\sim 0^+$

For the TM polarized input, the waveguide can propagate only one mode along the X-axis.

$SiO_2$  encapsulating film was also tried to prevent the  $Li_2O$  out-diffusion during the high temperature Ti-diffusion. To ensure a stoichiometric structure, 1000 Å thick  $SiO_2$  films were sputter coated on  $LiNbO_3$  substrates using a mixture of 2  $\mu m$   $O_2$  and 2  $\mu m$  Ar as the sputtering gas. The diffusion conditions remained unchanged. After 3-1/2 hours diffusion at 950°C, waveguides of one in-diffusion and three out-diffusion modes were formed. The out-diffusion mode patterns are shown in Fig. 3. The weak spot on the left is the single mode due to Ti in-diffusion. The  $SiO_2$  film dissolved readily in diluted HF etching solution. However, the removal of  $SiO_2$  film did not affect the waveguide modal properties.

Based on our experimental results we conclude that neither  $SiO$  nor  $SiO_2$  films can prevent  $Li_2O$  from escaping the  $LiNbO_3$  substrate surface. In fact,  $Li_2O$  and  $SiO_2$  may react to form some kind of glass structure which is very susceptible to HF etchant.

## 2. Temperature and Time Dependence of $LiNbO_3$ Powder Treatment

As a result of 1976 IR&D effort, we have developed a technique to eliminate  $Li_2O$  out-diffusion waveguide by annealing the crystal in  $LiNbO_3$  powder.<sup>32</sup> The mechanism of  $Li_2O$  compensation process was explained to be the following. At high temperature, the loosely bound lithium oxide molecules tend to escape from the surface. Since  $LiNbO_3$  powder has more surface area than  $LiNbO_3$  wafers, the  $Li_2O$  vapor pressure due to the powder is sufficient to build up a  $Li_2O$  rich environment so that the out-diffusion process from the  $LiNbO_3$  wafer is suppressed. Furthermore, the compensation process of diffusing  $Li_2O$  back into a lithium deficient  $LiNbO_3$  wafer becomes thermodynamically favorable.

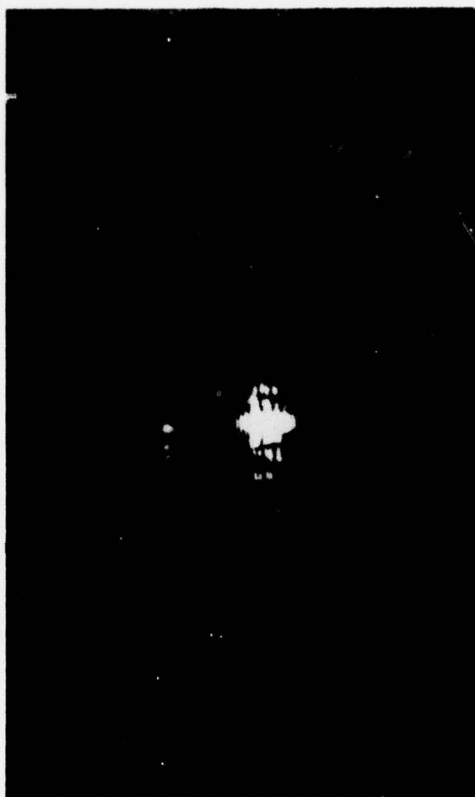


Figure 3.  
Li<sub>2</sub>O Out-diffusion Waveguide Modes  
for a SiO<sub>2</sub> Encapsulated Ti Diffusion  
in LiNbO<sub>3</sub>.



Under this program, the effect of  $\text{LiNbO}_3$  powder on the waveguide formation in  $\text{LiNbO}_3$  was investigated in a greater detail. A series of experiments was carried out to study the temperature and time dependence of this powder treatment. Three significant results were obtained. First, it is confirmed that up to 1% molecular weight of  $\text{Li}_2\text{O}$  can be brought into the substrate surface layer through the powder treatment. Second, the compensation process ( $\text{Li}_2\text{O}$  in-diffusion) may occur at a temperature  $750^\circ\text{C}$  or lower. Third, 80% of the compensation process can be obtained in one-half hour when the annealing temperature is  $900^\circ\text{C}$ . The fast reaction rate implies that the powder treatment may not be dominated by the diffusion process. Solid-solid surface reaction may play an important role in this process.

To characterize the  $\text{LiNbO}_3$  powder annealing process, we define a parameter  $\Delta n_e$  as

$$\Delta n_e = n_f - n_i$$

where  $n_i$  and  $n_f$  are the effective modal indices of TE modes before and after the  $\text{LiNbO}_3$  powder treatment. The modal indices are determined to within 0.001 by measuring the excitation angle of a rutile prism coupler. To study the temperature dependence, a planar waveguide was fabricated by diffusing  $200 \text{ \AA}$  Ti into a  $1 \text{ in}^2$  Y-cut  $\text{LiNbO}_3$  substrate. The diffusion was done in a flowing oxygen atmosphere and the diffusion temperature (T) and diffusion time (t) were  $T = 950^\circ\text{C}$ ,  $t = 16 \text{ h}$ . After diffusion, the waveguide can support two Ti in-diffusion modes and two  $\text{Li}_2\text{O}$  out-diffusion modes for TE polarization waves propagating along the X-axis. The index of refraction of the  $\text{TE}_0$  mode was 0.005 above that of the substrate. The sample was then cut into six smaller pieces; five of them were annealed in  $\text{LiNbO}_3$  powder at different temperatures. The annealing time was fixed at 16 h. After the powder annealing, the waveguide modal index was measured again with rutile prism couplers. It was noticed that all the in-diffusion waveguides became single mode in addition to the elimination of out-diffusion waveguides. Figure 4 shows the modal index change for  $\text{TE}_0$  mode as a function of the annealing temperature.  $\Delta n_e$  varies from

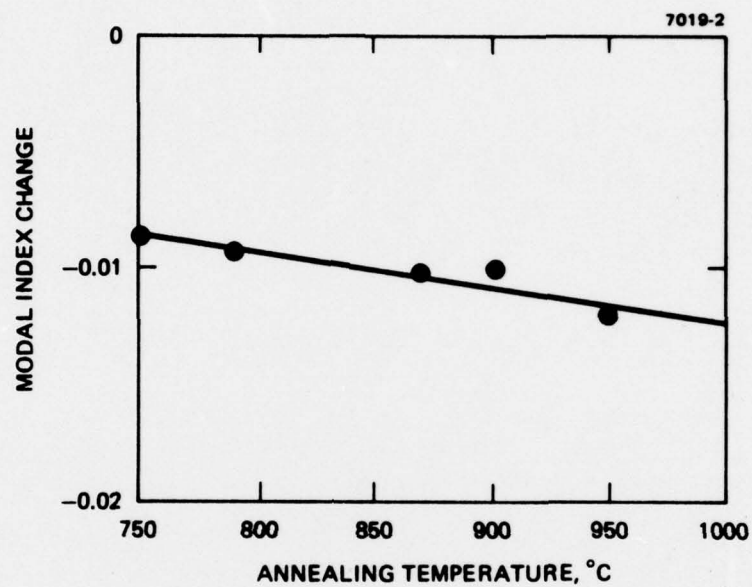


Figure 4. Modal Index Change for  $TE_0$  Mode as a Function of Temperature.

-0.01 to -0.012 as the temperature increases from 750°C to 950°C. The amount of change is even greater than that due to the Ti in-diffusion. Since the extraordinary refractive index of  $\text{LiNbO}_3$  increases approximately linearly as the  $\text{Li}_2\text{O}$  concentration decreases, an index decrease of 0.012 represents an increase in molecular weight concentration of  $\text{Li}_2\text{O}$  by 0.8%. The effective index of refraction for the TM mode did not show significant changes although the throughput of the prism coupling configuration had dropped by a factor of 3-5.

The next question to be addressed is how fast is this annealing process. One  $\text{LiNbO}_3$  waveguide was annealed in  $\text{LiNbO}_3$  powder at 900°C. The modal index was checked at several different annealing times. The results are shown in Fig. 5. Eighty percent of the maximum index change occurred in less than half an hour and the asymptotic value was approached after 2-1/2 hours.

### 3. A New Diffusion Source, Titanium Silica Film

During the search for diffusion sources other than e-beam evaporated (or RF sputtered) Ti metal, an organo-metallic liquid called TITANIUMSILICAFILM was brought to our attention. The film converts into  $\text{SiO}_2$  and  $\text{TiO}_2$  at a moderately high temperature. The liquid can be applied to  $\text{LiNbO}_3$  substrates in a thin-film form using dip-coating technique and  $\text{TiO}_2$  molecules in  $\text{SiO}_2$  glass matrix can be used as the diffusion source. The  $\text{TiO}_2 \cdot \text{SiO}_2$  proportion may vary by mixing TITANIUMSILICAFILM solution with appropriate amount of pure SILICAFILM solution. The solution we tried was solution B which contains 28%  $\text{SiO}_2$  and 72%  $\text{SiO}_2$  after conversion. At pulling speed 1/4 in/min, the resulting film thickness was about 1150 Å.

In the preliminary experiment under IR&D program, a Y-cut  $\text{LiNbO}_3$  was dip-coated with the TITANIUMSILICAFILM. The coating was very uniform except edges where thinner coating was obtained. The diffusion was carried out at 950°C for 16 hours. Before diffusion, the sample was baked at 600°C for 4 hours to ensure a complete conversion. The residue film on the surface can be etched away readily with diluted (~30%) HF. However, the area coated with TITANIUMSILICAFILM had been raised by about 200 Å. According to our experience with diffusion of e-beam evaporated Ti film,

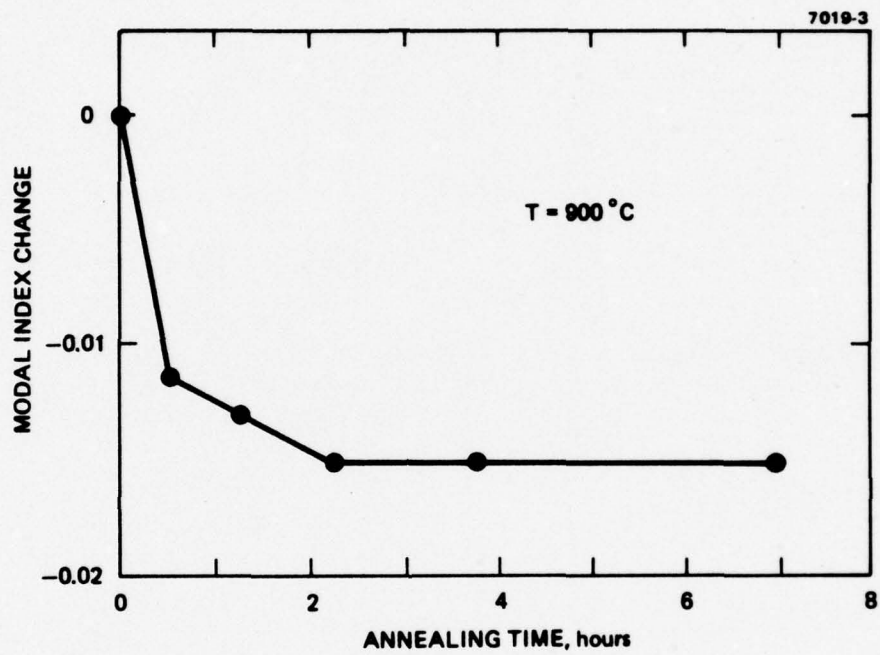


Figure 5. Modal Index Change as a Function of Annealing Time ( $T = 900^{\circ}\text{C}$ ).



the resulting diffusion area would raise by 2.5 times the original Ti film thickness. A 200 Å increase indicates that the amount of Ti metal diffused into the  $\text{LiNbO}_3$  substrate is equivalent to 80 Å thick Ti coating.

The resulting waveguide has good surface appearance. One TE mode and one TM mode have been successfully excited in the waveguide using a He-Ne laser and  $\text{TiO}_2$  prism couplers. The mode patterns for both polarizations are shown in Fig. 6. One thing not expected was that no  $\text{Li}_2\text{O}$  out-diffused waveguide was observed. This behavior is not currently understood. It might be that  $\text{TiO}_2$  and  $\text{SiO}_2$  composite film is denser and effectively eliminates the out-diffusion process. A detailed investigation on this new processing technique is underway.

The waveguide loss was measured using two prism couplers. The first prism couples TE polarization He-Ne laser light into the waveguide and the second prism extracts the guided light to a detector. By monitoring the output as a function of the prism separation the waveguide loss was determined from the slope of a semi-logarithmic plot. The experimental results are shown in Fig. 7. The waveguide loss was calculated to be 1.6 dB/cm. This loss number is slightly higher than for the diffusion waveguide obtained from diffusion of e-beam evaporated Ti. We expect comparable waveguide properties can be obtained with more careful processing and handling. According to the information supplied from the manufacturer, the impurity concentration of the solution are  $\text{Fe} < 1/2 \text{ PPM}$ ,  $\text{Cu} < 1 \text{ PPM}$  and  $\text{Na} < 1 \text{ PPM}$ . Hence, this organo-metallic solution will offer a new, low cost and high purity Ti source for waveguide fabrication.

### C. Lateral Diffusion Studies

At high temperature, the diffusion of Ti channels proceeds in both lateral (Z-axis) and downward (Y-axis) directions. The diffusion can be described by the second order partial differential equation

$$\frac{\partial N(y,z,t)}{\partial t} = D_y \frac{\partial^2 N(y,z,t)}{\partial y^2} + D_z \frac{\partial^2 N(y,z,t)}{\partial z^2}$$

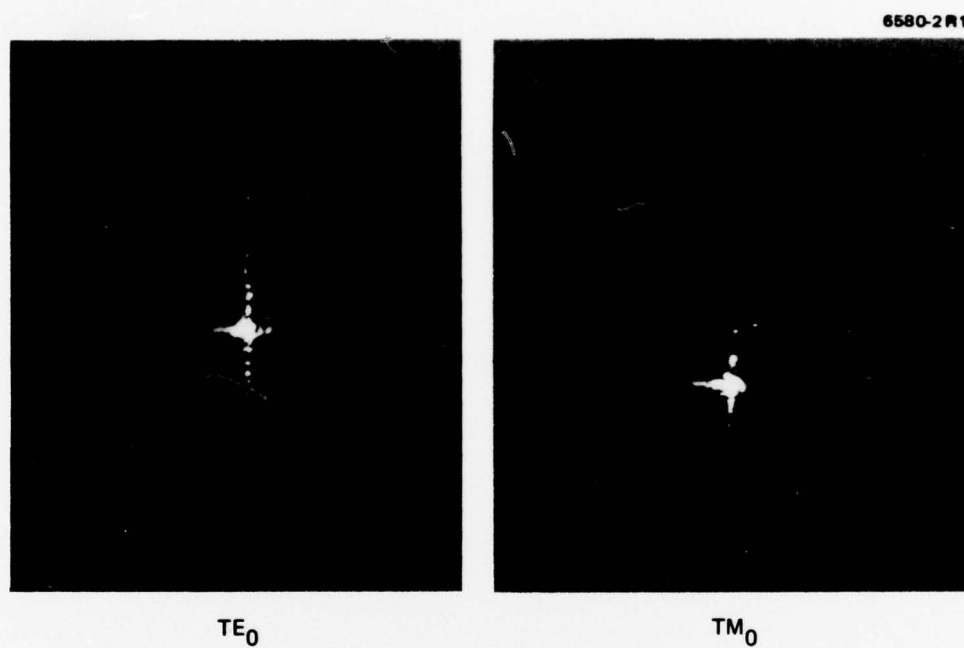


Figure 6. Single Mode Waveguide Formed in  $LiNbO_3$  by Diffusing Ti Doped Silica.

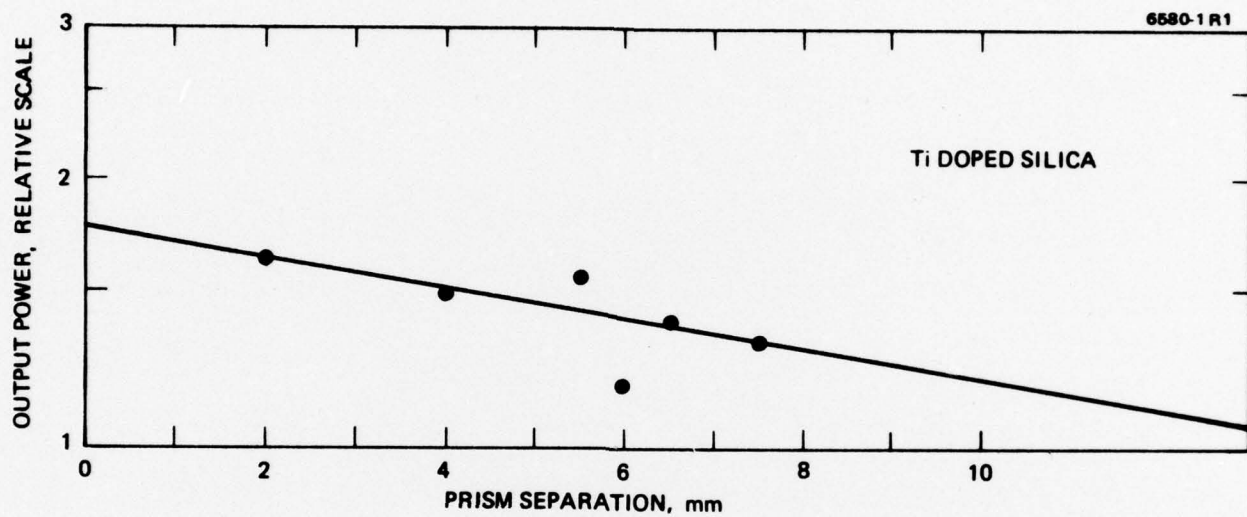


Figure 7. Loss Measurement for Single Mode  $\text{LiNbO}_3$  Waveguide Fabricated by Diffusing Ti Doped Silica.

where  $D_y$  and  $D_z$  are diffusion coefficients along  $y$  and  $z$  axes respectively. The magnitude of  $D$  gives a measure of the relative ease or difficulty with which the diffusant can move about in its environment.  $\text{LiNbO}_3$  and  $\text{LiTaO}_3$  are uniaxial crystals with the  $c$ -axis along the  $z$  direction. It has been reported that an enhanced lateral diffusion, at a rate of about 20 times the downward diffusion rate, was observed in Nb diffused  $\text{LiTaO}_3$  channel waveguides.<sup>4</sup> We have studied the Ti concentration profile near the edge of a strip of 70  $\mu\text{m}$  initial width using electron microprobe technique.<sup>7</sup> No enhanced lateral diffusion was observed.

During this program, the lateral diffusion profile of 4  $\mu\text{m}$  wide single mode channel waveguide was measured using the same microprobe technique. A focused electron beam was scanned across the sample surface at an angle of  $32^\circ$  with respect to the channel edge. The concentration of Ti was monitored by the intensity of emitted characteristic X-ray emission of Ti. The penetration depth of the electron beam is estimated to be about 1  $\mu\text{m}$  at the beam energy of 20 keV. Using one-dimension diffusion model, the  $Z$ -dependence of diffusion profile is given by

$$N(z) = \frac{N_0}{2} \left\{ \text{erf} \frac{h-z}{2\sqrt{D_z t}} + \text{erf} \frac{h+z}{2\sqrt{D_z t}} \right\}$$

where  $N_0$  is the initial surface concentration of Ti channel and  $2h$  is the initial channel width. Figure 8 shows the results of electron microprobe measurement and a plot of the theoretical function  $N(z)$ . The diffusion time was 4 h and the diffusion coefficient along the  $Z$ -axis is assumed to be

$$D_z = 2 \times 10^{-8} \text{ cm}^2/\text{hour}$$

The measurement is in good agreement with the theoretical curve except at tails where the measured concentration is higher. The difference could be due to the finite resolution ( $\sim 1 \mu\text{m}$ ) of electron microprobe technique. The half maximum width is about 10  $\mu\text{m}$  as compared to the original Ti channel width of 4  $\mu\text{m}$ . The lateral diffusion on either side is about 3  $\mu\text{m}$ ; this is comparable with the downward diffusion depth.



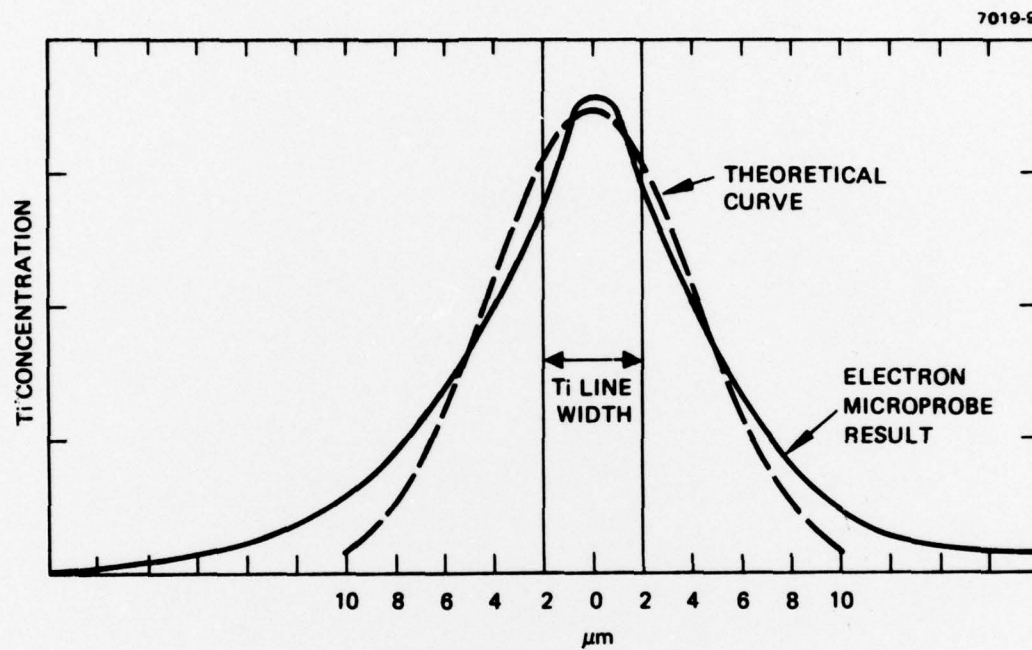


Figure 8. Lateral Diffusion Profile of a Ti Channel of 4  $\mu\text{m}$  Initial Width.

The equal diffusion rates along the y and z axes was further verified by the results of mode intensity profile measurement of the single mode channel waveguide. The mode was excited by an end-fire arrangement. The input He-Ne laser light was focused onto the edge of the channel guide. The near field pattern was then magnified by a 50 X microscope objective and scanned with a slit and a detector. Figure 9 shows the results of scan along the depth and width directions. The FWHM are  $1.8\text{ }\mu\text{m}$  and  $4.4\text{ }\mu\text{m}$ , respectively. Because of micron size chips at the edge, the measured (dot) points scattered around the Gaussian-like curve in Fig. 9(b).

### III. FORMATION OF THREE-DIMENSIONAL CHANNEL WAVEGUIDE HORNS

Usually, single-mode channel waveguides used in optical circuits require shallow waveguide depth to obtain a large interaction between guided optical field and surface electric or acoustic field. The typical cross section of a diffused channel waveguide is about 4 by  $2\text{ }\mu\text{m}$ , while single mode optical fibers have relatively larger core diameters ranging from 5 to  $15\text{ }\mu\text{m}$ . To assure a good mode matching in optical fields, we can use either a small core fiber or a three-dimensional horn structure at the end of the channel waveguide. Using small core fiber in an optical system is not practical because of the severe coupling tolerance requirements. On the contrary, the combination of three dimensional horn structure and a relatively large core fiber will make the tolerance less critical. During the program, we have developed a novel technique for forming the three-dimensional horn structure; it uses a controlled  $\text{CO}_2$  laser heating.

In principle, the formation of a horn-like structure is determined by a number of parameters such as laser spot size, absorption coefficient of  $\text{LiNbO}_3$  at  $10.6\text{ }\mu\text{m}$ , thermal conductivity, temperature dependence of the Ti diffusion coefficient, and diffusion time. The mathematical theory of diffusion in isotropic medium is based on the hypothesis that the rate of transfer of the diffusion substance through unit area of a section is proportional to the concentration gradient in

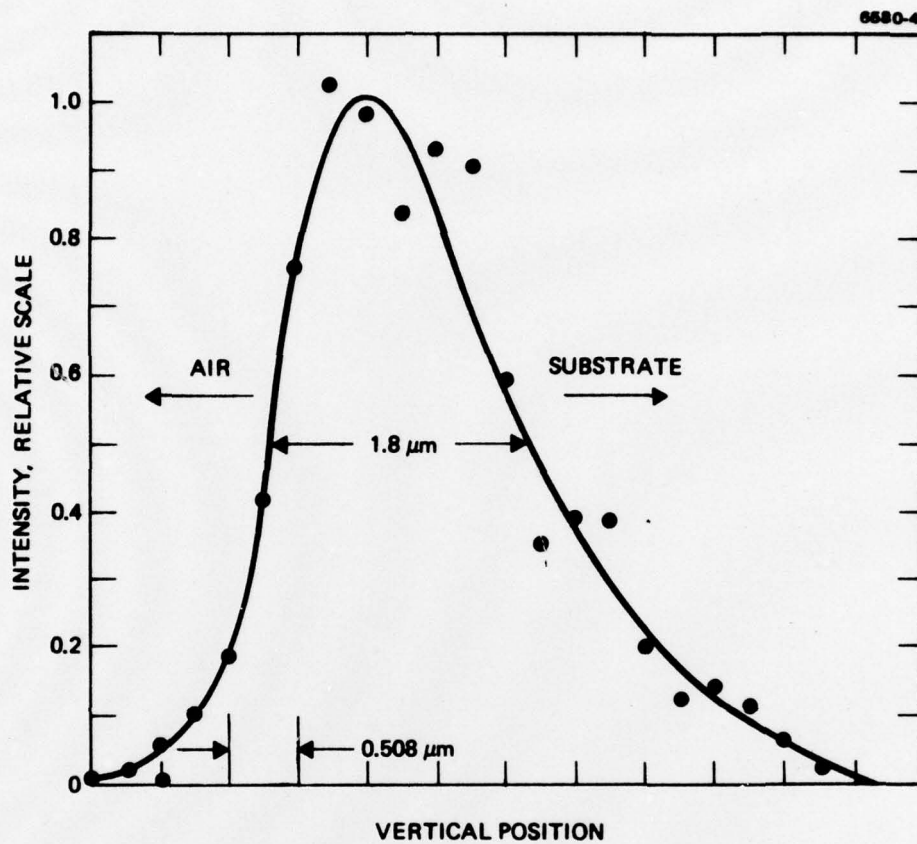


Figure 9(a). Intensity Profile of a 4  $\mu\text{m}$  single mode channel Waveguide along the Depth Direction.

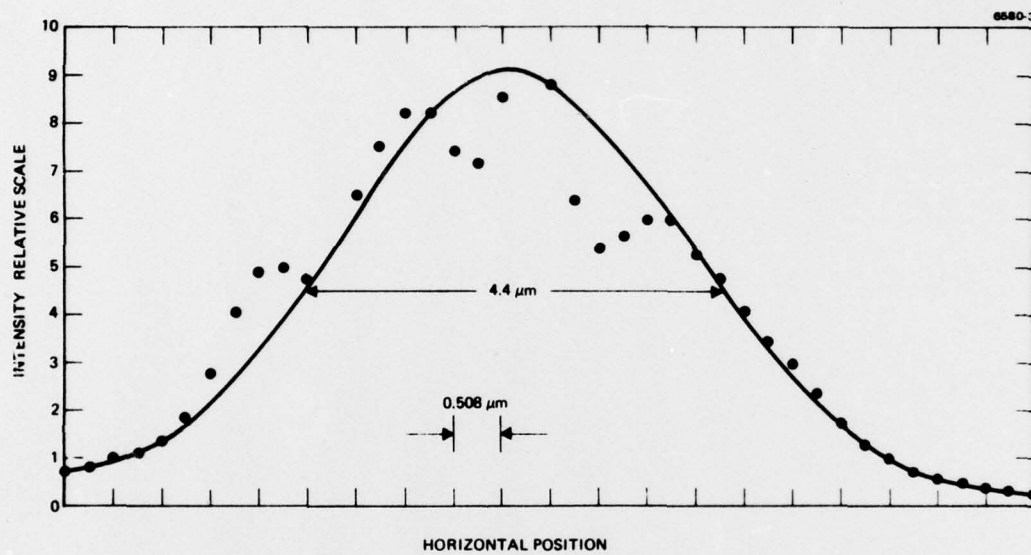


Figure 9(b). Intensity Profile of a 4 μm Single Mode Channel Waveguide along the Width Direction.



the direction normal to the section, or  $F = -D\partial N/\partial x$ , where  $F$  is the rate of transfer per unit area,  $N$  is the concentration of the diffusion substance, and  $D$  is the diffusion coefficient. The negative sign arises because the diffusants move from a high to a lower concentration region. The magnitude of  $D$  gives a measure of the relative ease or difficulty with which the diffusion substance can move about in its environment. For the diffusion of substitutional impurities, such as Ti diffusion in  $\text{LiNbO}_3$ , the diffusion coefficient has its temperature dependence given by  $D = D_0 \exp(-E_a/kT)$ , where  $k$  is Boltzmann constant and  $E_a$  is defined as activation energy. For the diffusion process induced by  $\text{CO}_2$  laser heating, the sample temperature and thus the diffusion coefficient vary from point to point. Assuming the activation energy is 2.7 eV,<sup>8</sup> the diffusion rate at  $T = 850^\circ\text{C}$  is almost 15 times the diffusion rate at  $T = 750^\circ\text{C}$ . The differential equation of diffusion is given by

$$\frac{\partial N}{\partial t} = \frac{\partial}{\partial x} \left( D_x \frac{\partial N}{\partial x} \right) + \frac{\partial}{\partial y} \left( D_y \frac{\partial N}{\partial y} \right) + \frac{\partial}{\partial z} \left( D_z \frac{\partial N}{\partial z} \right)$$

The solution of Ti concentration distribution and hence the horn structure can only be obtained by numerical computing technique.

Consider the case of a CW laser beam with a Gaussian profile absorbed at the surface of a semi-infinite solid. Assuming no radiation loss, the temperature increase,  $\Delta T$ , at the center of the irradiated area can be calculated by

$$\Delta T = \frac{P}{2\sqrt{\pi} dK}$$

where  $K$  is thermal conductivity,  $P$  is the total power absorbed, and  $d$  is the Gaussian beam size at  $(1/e)^2$  point.<sup>9</sup> If we substitute numbers into this equation for a  $\text{LiNbO}_3$  wafer with a thermal conductivity of  $4.2 \times 10^{-2} \text{ W cm}^{-1} \text{ deg}^{-1}$  irradiated with a 2W CW  $\text{CO}_2$  laser of spot size  $d = 0.5 \text{ mm}$ , the temperature increase at the center is about  $270^\circ\text{C}$ .

$\text{LiNbO}_3$  is a brittle material. The thermal expansion coefficient in the  $a$ -axis is  $16.7 \times 10^{-6}$  per  $^\circ\text{C}$  over the range from room temperature to  $600^\circ\text{C}$  and the  $c$ -axis coefficient is  $2 \times 10^{-6}$  per  $^\circ\text{C}$  over the range from room temperature up to  $600^\circ\text{C}$ .<sup>10</sup> To avoid thermal cracking due to a large thermal gradient it is important to know what the maximum

temperature difference that can be imposed on  $\text{LiNbO}_3$  crystals. Experimentally, it is difficult to measure the temperature profile at the laser irradiated area. A simple test was designed to determine indirectly what the maximum temperature difference is that the  $\text{LiNbO}_3$  wafer can take without cracking. Y-cut  $\text{LiNbO}_3$  wafers of thickness 1/2 mm were irradiated with  $\text{CO}_2$  laser beam with spot size 500  $\mu\text{m}$ . Although some samples could absorb up to 5 W of laser power without cracking, all the diffusion experiments were done at a power level 2 W or less. When a Ti (200 Å) coated  $\text{LiNbO}_3$  wafer was heated with 2 W  $\text{CO}_2$  laser, oxidation of the Ti began at furnace temperatures as low as 350°C. It is known that Ti oxidizes at a temperature around 500°C. This result indicates that the temperature increase due to laser heating was about 150°C.

A 3" high vertical furnace was constructed to provide a uniform heating of  $\text{LiNbO}_3$ . The  $\text{CO}_2$  laser beam for enhanced local heating was directed onto the sample holder in the furnace by a steering mirror. During the heating experiment, the furnace temperature was maintained at  $T = 650\text{--}850^\circ\text{C}$  and the laser power was varied from 1.2 to 3.0 watts. Surface damage was always the major problem partially due to the fact that diffusion was performed in air. There are speckles and etched pits at laser irradiated area as shown in Fig. 10(a). The sample was heated with 1.2 W laser for 20 h. and the furnace temperature was 760°C. The three vertical lines are 4  $\mu\text{m}$  wide single mode channel waveguides. Most etched pit clustered around surface scratches while speckles were scattered over the heated area. However after annealing the sample in oxygen atmosphere for one hour at 850°C, most speckles were annealed away, Fig. 10(b). In phase II of this program, the  $\text{CO}_2$  laser enhanced diffusion will be carried out in an oxygen atmosphere. The diffusion furnace will be redesigned to include an oxygen flowing system.

Experimental results showed that 50% increase in diffusion rate was obtained for one channel waveguide. The diffusion parameters were  $P_{\text{CO}_2} = 1.25$  W,  $T = 850^\circ\text{C}$  and  $t = 4.5$  h. Oxygen annealing was done at  $T = 850^\circ\text{C}$  for one hour. Figure 11 shows pictures of guided modes for expanded and unexpanded channel waveguides. Figures 12 and 13 are the

near field intensity scan along the depth and the width directions. It is not known why the intensity distribution oscillates in the depth direction. More work is required to evaluate this expanded channel horn structure. Electron microprobe technique shall be used to actually plot out the Ti concentration profile.

#### IV. SUGGESTED PLANS FOR PHASE II

1. Fabrication of single mode channel waveguide without the presence of  $\text{Li}_2\text{O}$  out-diffusion waveguide. Thicker coating of Ti and/or lower  $\text{Li}_2\text{O}$  vapor pressure will be used.
2. Fabrication and evaluation of three dimensional channel horn structures by  $\text{CO}_2$  laser localized heating. The diffusion will be performed in oxygen atmosphere.
3. Study of the end-butt coupling of single mode optical fiber and expanded single mode channel waveguide. More than 60% coupling efficiency has been achieved from single mode channel waveguide to single mode optical fiber. The optical fiber has core diameter 12  $\mu\text{m}$  and is single mode for optical wavelengths greater than 0.85  $\mu\text{m}$ .



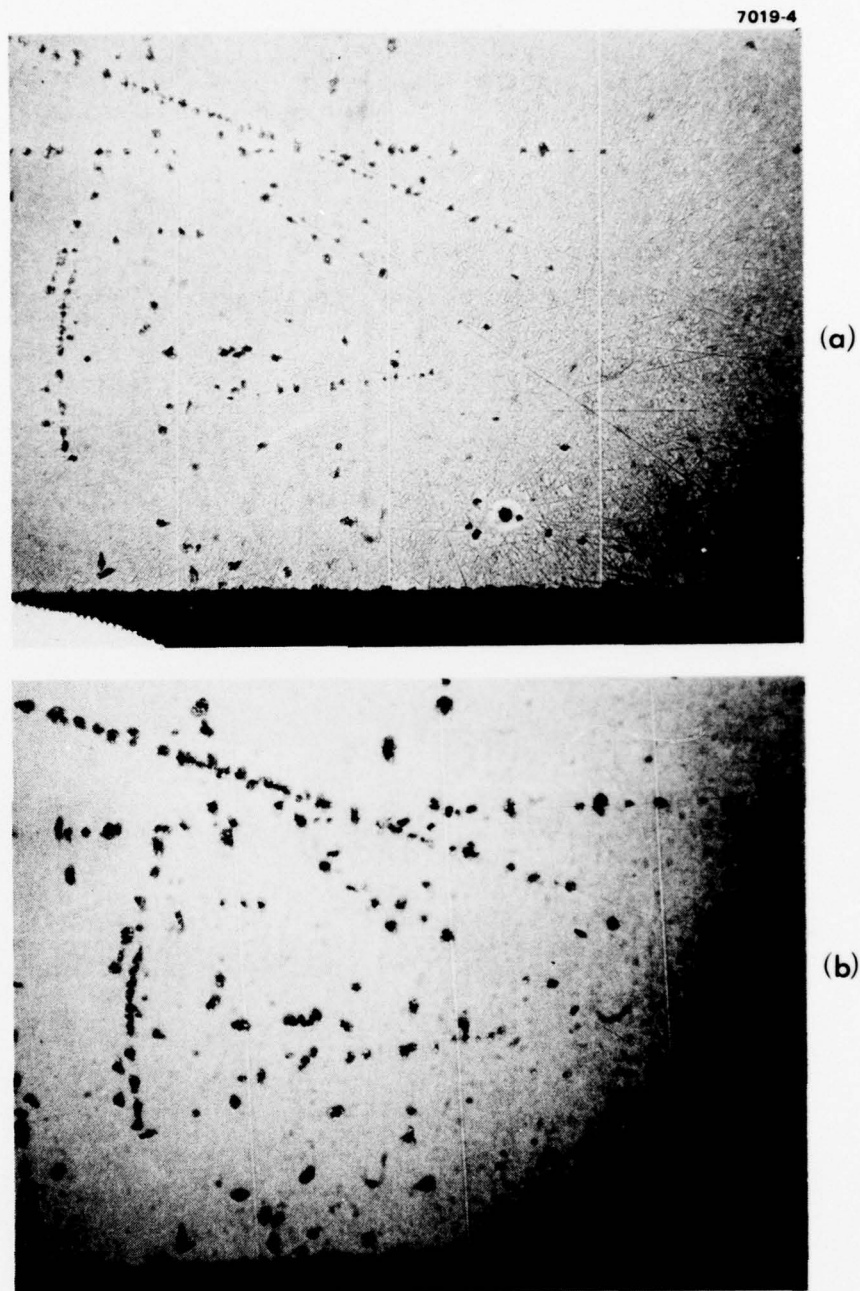
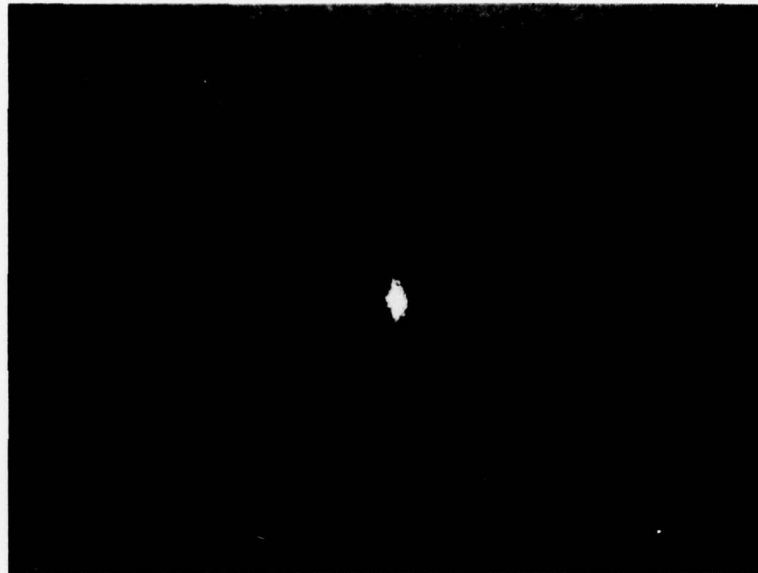


Figure 10. (a) Surface Damage due to  $\text{CO}_2$  Laser Heating.  
(b) Surface Structure after 1 h Annealing  
in Oxygen.



7019-6

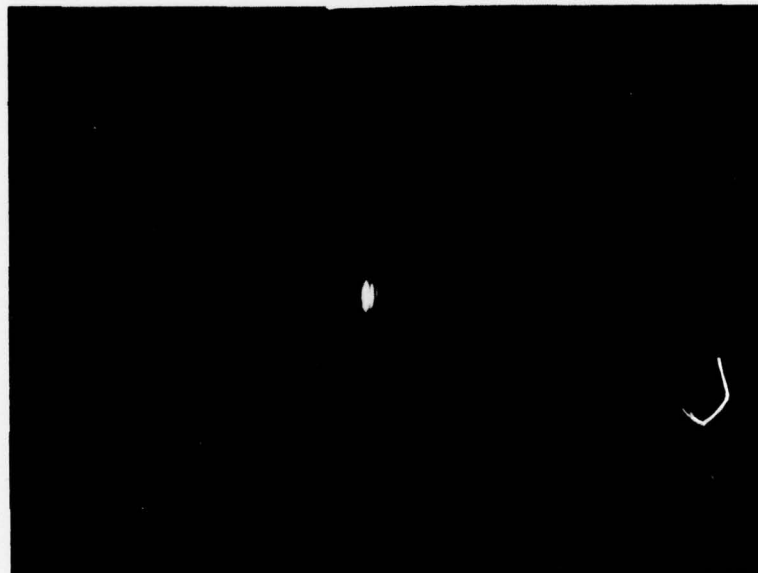


(a)

AIR



SUBSTRATE



(b)

Figure 11. Near Field Intensity Distribution for (a) Expanded and (b) Unexpanded Channels.

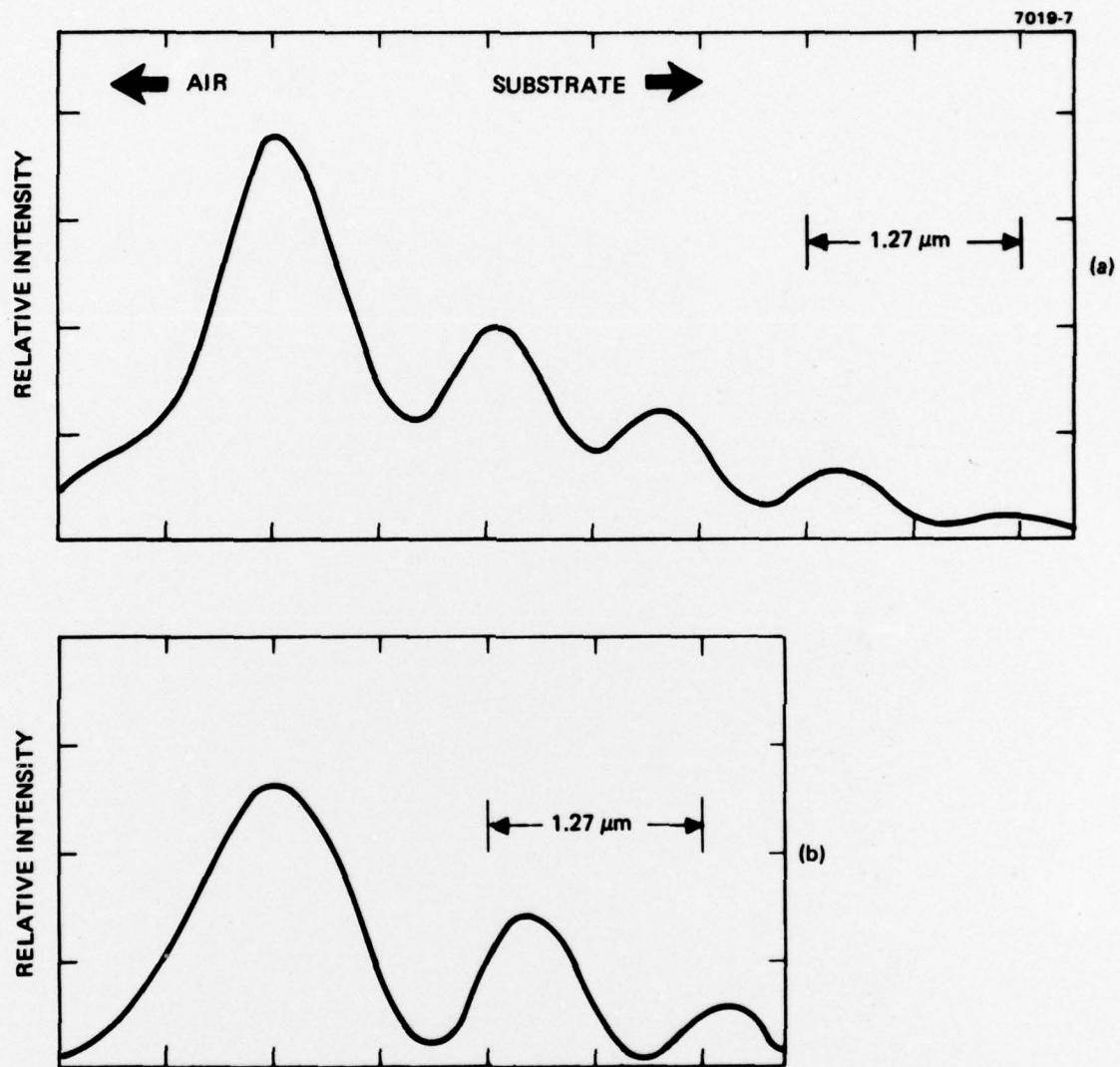


Figure 12. Intensity Scan along the Depth Direction for (a) Expanded and (b) Unexpanded Channel Guides.

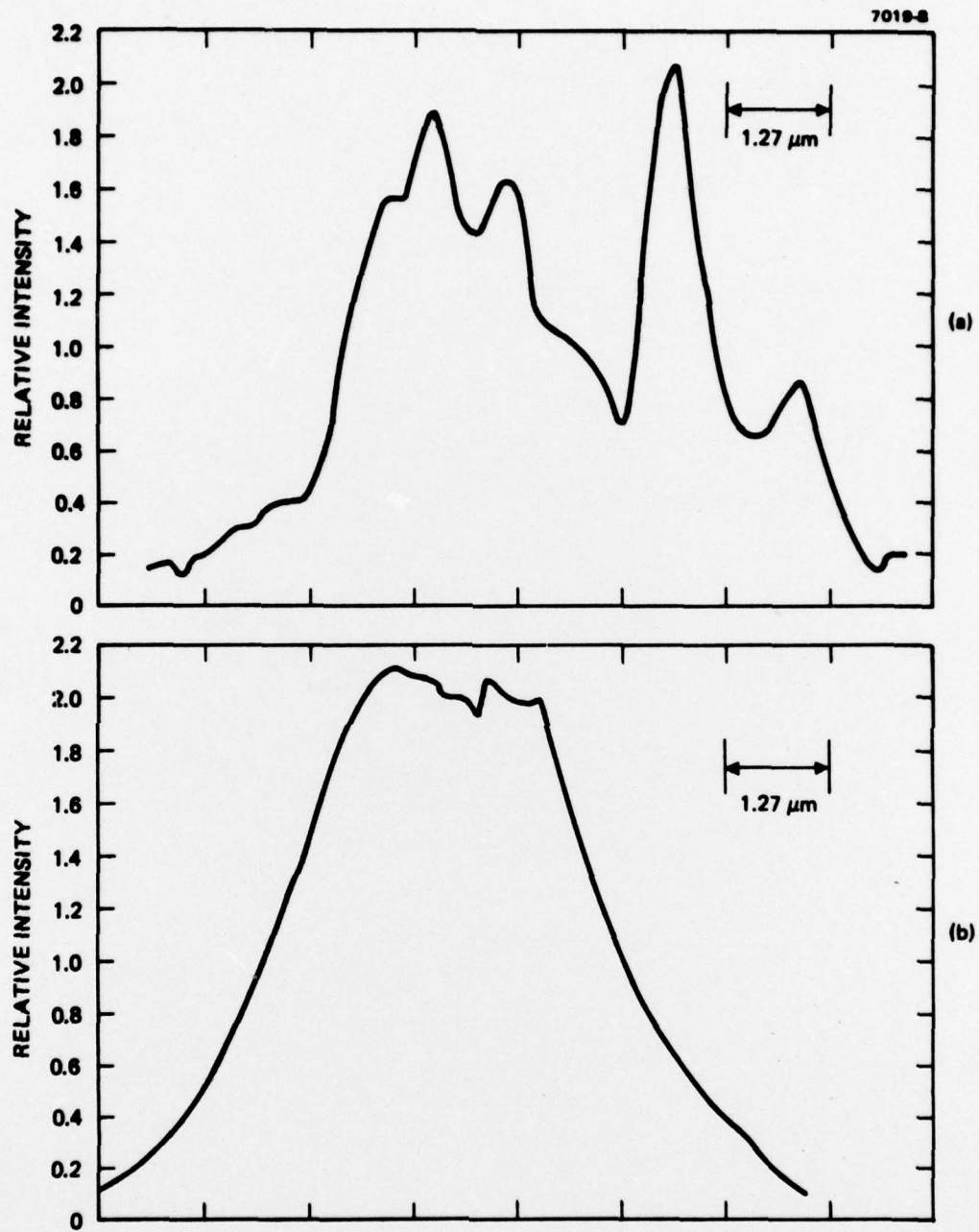


Figure 13. Intensity Scan Along the Channel Width Direction for (a) Expanded and (b) Unexpanded Channel Guides.

## REFERENCES

1. R. V. Schmidt and I. P. Kaminow, Appl. Phys. Lett. 25, 458 (1974); J. M. Hammer and W. Phillips, Appl. Phys. Lett. 24, 545 (1974).
2. I. P. Kaminow and J. R. Carruthers, Appl. Phys. Lett. 22, 326 (1973).
3. B. Chen and A. C. Pastor, Appl. Phys. Lett. 30, 570 (1977).
4. J. M. Hammer, W. Phillips, and C. C. Neil, "Channel Waveguide Study," Contract N00014-75-C-0078, January 1978.
5. H. P. Hsu and A. F. Milton, Electronics Lett. 12, 404 (1976); W. K. Burns and G. B. Hocker, Appl. Opt. 16, 2048 (1977).
6. L. Goldberg and S. H. Lee, Int'l. Conf. on Optical Fibers and Integrated Optics, July 18, 1977, Tokyo, Japan.
7. B. Chen and G. L. Tangonan, "Thin-Film Optical Switch," Final Report, Feb. 1977, Contract Number N00173-76-C-0113.
8. It. Naitoh, M. Nunoshita and T. Nakayama, Appl. Opt. 16, 2546 (1977).
9. J. F. Ready, "Effects of high-power laser radiation," Academic Press, 1971.
10. S. C. Abrahams, H. J. Levinstein and J. M. Reddy, J. Phys. Chem. Solid. 27, 1019 (1966).